10.3 Secondary standard buffer 7.00 (available through commercial vendors).

10.4 Potassium chloride (KCl) - reagent grade or higher

10.5 Probe Storage Solution, 3.5M KCl

10.5.1 Dissolve 52.19 g KCl (Section 10.4) into 200 ml of reagent water.
10.5.2 Alternatively, add 1g KCl to 200ml pH 4 buffer for probe storage.
10.5.3 Change this solution weekly.

10.6 Potassium hydrogen phthalate (KHP), reagent grade or higher

10.7 KHP Solution - 0.05M

10.7.1 Add 10.12g of dry KHP to approximately 500 ml of DI water.
10.7.2 Bring to final volume of 1L
10.7.3 Transfer to an appropriately labeled bottle.
10.7.4 This solution is stable for 1 month or until degradation is noted. The expiration may not exceed that of its parent.

11) Method Calibration

11.1 Meter Calibration

11.1.1 Ensure the pH probe fill hole is open to atmospheric pressure if using a refillable probe.
11.1.2 Follow the manufacturer's instructions for pH meter calibration.

11.2 Mettler Toledo Five Easy Plus Calibration

11.2.1 Select a predefined buffer group (Initial setup and following power failure)

11.2.1.1 Press and hold the MODE/SETUP key until the setup icon appears on the display and the MTC temperature blinks.
11.2.1.2 Press READ to ignore, the current temperature unit blinks, press READ to ignore again.
11.2.1.3 When the current buffer group blinks, the buffers belonging to this buffer group appear alternating on the display.
11.2.1.4 Press the up or down arrow to select another buffer group and press READ to confirm your selection.
11.2.1.5 The meter will automatically exit to the measurement screen.

11.2.2 Place the electrode in the 1.68 buffer solution and press "CAL"
11.2.3 Allow the reading to stabilize, indicated by the stabilization icon and press "READ". The 1.68 buffer value is displayed and stored.
11.2.4 Rinse the electrode with DI water.
11.2.5 Place the electrode in the 4.01 buffer solution and press "CAL"
11.2.6 Allow reading to stabilize and press "READ"
11.2.7 Rinse the electrode with DI water.
11.2.8 Place the electrode in the 10.01 buffer solution and press "CAL"
11.2.9 Allow reading to stabilize and press "READ"
11.2.10 Press "READ" again to store the calibration; the slope will be displayed for 3 seconds. Record the slope.
11.2.11 An acceptable slope for the Electrode is 95-105%. A slope of 90-94% indicates the probe may require maintenance. A slope below 90 indicates the probe shall be replaced.
11.2.12 To reject the calibration and start over, press "EXIT" during the 3 second

display of the slope.

11.3 SPER Scientific pH Calibration

11.3.1 Press "POWER" to turn the meter on and "MODE" continuously to select pH.
11.3.2 Rinse the probe with DI. Shake and air dry, but do not wipe the probe dry.
11.3.3 Select the 1.68 pH buffer and pour solution into a clean container (sufficient volume to immerse the probe tip).
11.3.4 Immerse the probe tip and gently stir to create a uniform sample.
11.3.5 Press "CAL" to enter calibration mode. "CAL" will flash on the lower left.
11.3.6 The main value indicates the measured value, and the secondary value indicates the desired buffer value.
11.3.7 A custom range is selected, so press "HLD" to select the buffer range needed. Press the up or down arrows to adjust the lower middle display to coincide with the main display reading.
11.3.8 When the measured pH value is stable "READY" will appear on the left side of the screen. Press to confirm.
11.3.9 Repeat steps 11.3.2 - 11.3.8 for the 4.01 and 10.01 buffers.
11.3.10 After the final buffer has been calibrated, press "ESC"
11.3.11 To view the slope, press "MODE" to select the probe type as pH.
11.3.12 Press "SET" for 2 seconds.
11.3.13 Press up arrow until "ELE" appears in the middle display and P3.0 appears in the lower display.
11.3.14 Press "enter arrow" to enter P3.1. The screen displays one of four available slope values. Record each slope in the logbook

11.3.14.1 Each slope must fall between 75-115%.
11.3.14.2 If outside criteria, replace the probe and repeat the calibration process.

11.4 VWR Symphony Calibration

11.4.1 Place the electrode in the 4.01 buffer solution and press the "CAL" button.
11.4.2 The meter will accept the reading as soon as the measurement is stable.
11.4.3 Rinse the electrode with reagent water and press the "CAL" button and repeat using the 7.00 buffer solution.
11.4.4 Rinse the electrode with reagent water and press the "CAL" button and repeat using the 10.01 buffer solution.
11.4.5 Press the "MEAS" button when the reading is stable. The slope will then flash on the screen. Record the slope in the pH logbook.

11.4.5.1 Slope must fall between 95-105%.
11.4.5.2 If outside criteria, repeat the calibration process.

11.5 After calibration is complete, a secondary standard 7.00 buffer must be used as the initial check standard. Acceptance criteria are a pH of 6.95 - 7.05. If the check standard fails, the meter must be recalibrated.

11.6 Perform a secondary check (or LCS) with the KHP solution (Section 10.7).

11.7 Record the buffer BPL identifiers in the associated bench logbook.

11.8 The pH meter must be calibrated daily and verified (or re-calibrated) after every twenty (20) samples.

12) Sample Preparation/Analysis

12.1 Lab Control Sample

12.1.1 Determine the pH of an aliquot of the 0.05M KHP solution and report as the LCS.

12.2 Sample Preparation (solid samples):

12.2.1 Thoroughly homogenize an aliquot of sample large enough for the analysis.
12.2.2 Prepare a 1:1 reagent water extract by weighing 20g of homogenized sample and adding 20 ml of reagent water.
12.2.3 Record amount of solid used in the LIMS prep batch. Change the final volume to maintain the 1:1 proportion of solid:water accordingly.
12.2.4 If the solid matrix consumes all the aqueous phase, add an additional 20 ml of reagent water.
12.2.5 Stir the sample continuously for five minutes on a stir plate with a stir bar.
12.2.6 Let the soil suspension sample stand for 1 hour to settle out the suspended particles. Alternatively, the sample may be filtered with the filtrate retained for analysis.
12.2.7 Continue with section 12.3.2 of Sample Analysis.

12.3 Sample Analysis:

12.3.1 Apply continual gentle mixing to the aqueous sample throughout the measurement.
12.3.2 Ensure the pH probe fill hole is open to atmospheric pressure if using a refillable probe
12.3.3 Immerse the electrode in an aliquot of the sample. For soils, make sure the probe is in the water column and is not in contact with the sediment layer.
12.3.4 Determine the sample pH. The meter will indicate a stabilized reading as soon as the measurement is stable.
12.3.5 Record the pH value directly from the meter.
12.3.6 Rinse the electrode with reagent water.
12.3.7 For aqueous samples, repeat the measurement on a successive aliquot of sample. Repeat this procedure until sample values differ by <0.1 pH units.
12.3.8 If a sample cannot be read on the meter, the unit will flash "9999" or "Error". If this occurs, use pH paper to gain an approximate pH value.
12.3.9 The use of pH paper is also recommended when the values bounce around and do not demonstrate stabilization or a sample is expected to cause harm to the pH probe.

12.4 Probe Storage

12.4.1 When not in use, ensure the probe fill hole is covered with the supplied sleeve.
12.4.2 Store the probe in pH electrode storage solution (10.5).

13) Troubleshooting

13.1 Make sure the electrode is filled with the appropriate solution (if applicable)

13.2 To remedy an abrupt change in slope try the following maintenance items:

13.2.1 For fat or oil build-up, degrease the membrane with cotton wool soaked in either acetone or a soap solution.

13.2.2 If the sensor membrane has dried out, soak the tip of the electrode in 0.1 M HCl overnight.

13.2.3 If a protein build-up has occurred in the diaphragm, remove deposits by soaking the electrode in a HCl/pepsin solution (commercially available)

13.2.4 If a silver sulfide contamination has occurred, remove deposits by soaking the probe in a solution of thiourea (commercially available)

13.3 For other items, refer to specific hardware manual.

14) Data Acquisition

14.1 All data must be recorded in the pH logbook. Batch header information must be filled out completely.

14.2 Aqueous samples should be qualified in the LIMS batch with the "SR01" qualifier in order to indicate potential bias due to the hold time deviation.

14.3 Samples with a pH > 10 should be qualified with the "pH01" qualifier in the LIMS batch as to potential bias if a low-sodium electrode is not used in the measurement process.

15) Calculation, and Data Reduction Requirements

15.1 Temperature correction is applied automatically by the pH meter. Results are automatically corrected to 25°C.

16) Quality Control, Acceptance Criteria and Corrective Action

16.1 Lab Control Samples

16.1.1 Frequency

16.1.1.1 Analyze a LCS with each analytical batch of 20 or fewer samples.

16.1.2 Acceptance

16.1.2.1 The analyzed concentration of the LCS must be within ±10% of the value documented in the applicable LIMS test code.

16.1.3 Corrective Action

16.1.3.1 Determine and correct the source of the deviation.

16.1.3.2 All samples associated with a failed LCS must be re-analyzed.

16.2 Sample Duplicate (Aqueous Analyses)

16.2.1 Frequency

16.2.1.1 The duplicate result for each aqueous sample is accomplished through the successive reading of an additional aliquot(s) of parent sample.

16.2.1.2 A duplicate result must be reported at a 10% frequency of analysis.

16.2.2 Acceptance

16.2.2.1 The duplicate must be within ±0.1 pH units from the parent analysis.

16.2.3 Corrective Action

16.2.3.1 Continue to analyze successive aliquots until the ±0.1 pH unit criteria is achieved.

16.2.3.2 If stabilization does not occur within the analysis of 3 subsequent aliquots, narrate as to potential bias due to analytical drift.

16.3 Sample Duplicate (Solid Analyses)

16.3.1 Frequency

16.3.1.1 The duplicate result for each solid sample is accomplished through the extraction and analysis of an additional aliquot of parent sample.

16.3.1.2 A duplicate result must be reported at a 10% frequency of analysis.

16.3.2 Acceptance

16.3.2.1 The duplicate must be within ±0.5 pH units from the parent analysis.

16.3.3 Corrective Action

16.3.3.1 Narrate as to potential bias due to improper sample homogenization.

16.4 Deviations and non-conforming events must be documented using a Nonconformance Corrective Action Report (NCAR) or as an Exception Report item on the laboratory review checklist. For mandatory QC failures (e.g. LCS), the NCAR must be submitted to the QA Manager via the NCAR database.

17) Data Records Management

17.1 All data is stored both electronically and hard copy for 10 years.

17.2 All analytical sequence IDs and standard preparation information must be recorded in the Run logbook. Hardcopy computer printouts of analytical sequences and raw data must be retained and initialed by the analyst (electronic initials are acceptable). To simplify standard tracking, analyst must attempt to use one lot of reagents and standards with each batch.

17.3 Complete all pertinent sections in the respective logbooks. If not-applicable then line out the section. "Z" out or "X" out all large sections of the worksheet that are not used. Make all corrections with single line through, date and initial. Make NO obliterations when manually recording data.

17.4 Logbooks are controlled. Never remove a page from a logbook. Completed logbooks are returned to the QA department when filled and no longer needed in the work area.

17.5 The effective date of this SOP is the date in the header or last signature date, whichever is most recent

18) Contingencies for Handling Out of Control Data

18.1 When method required QC exceedances occur, in every case where sample data quality

are affected, the source of the QC exceedance must be determined, corrected and sample reanalysis carried out when possible.

18.2 When affected sample analysis cannot be repeated due to limitations (i.e. sample availability, or if reanalysis can only be performed after expiration of a sample hold time), the reporting of data associated with exceeded QC data must be appropriately flagged and narrated. This documentation is necessary to define for the data user the effect of the error has upon the data quality of the results reported (e.g. E flag data indicate the result to be only an estimate).

18.3 All analysts must report sufficient comments in laboratory data review checklist for exceeded QC associated with sample results so that project management can further narrate and ensure data qualifiers (flags) are properly assigned to the reported data.

18.4 NCARs must be issued for QC system exceedances. Matrix interferences are reported using the analyte reporting comment section in LIMS or using the Laboratory Data review checklist.

18.5 Logbooks must be reviewed by the department supervisor monthly.

18.6 Logbooks must be reviewed by the QA staff quarterly.

19) Method Performance

19.1 Initial Demonstration of Proficiency- Each analyst must perform an initial demonstration of proficiency on a method and matrix basis with a successful analysis of four LCS where acceptable precision and accuracy are generated. The accuracy component must fall within LCS criteria. The precision component must be less than 20% for duplicate RPD data.

19.2 Method Detection Limits (MDLs) must be determined on an annual basis (at minimum) or whenever major modifications are performed.

19.3 Method performance must be verified every six months through participation in performance evaluation studies.

20) Summary of Changes

Table 20.1 Summary of Changes

Revision Number	Effective Date	Document Editor	Description of Changes
R05	8/15/13	CES	Formatting & Procedural changes in response to external audit findings.
R06	9/15/13	CES	Updated KHP solution prep and calibration procedure to include 4.00 point.
R07	02/15/15	CES	Addition of Calibration procedure for the Mettler Toledo meter.
R08	9/15/16	CES	Added section 1.3 for corrosivity characterization.
R08	9/15/16	CES	Added section 12.2.4 for additional water if needed.
R09	10/15/17	CES	Removal of cover page graphics
R09	10/15/17	CES	Significant update detailing use of the SPER pH Meter and update of slope acceptance criteria for each probe in use.
R09	10/15/17	CES	Updated method reference

21) References and Related Documents

21.1 U.S. Environmental Protection Agency, Methods for Chemical Analysis of Waters and Wastes, EPA/600/4-79-020, Method 150.1.

21.2 Standard Methods for Examination of Water and Wastewater 4500H-B, Online Edition, 2011.

21.3 Test Methods for Evaluating Solid Waste Physical/Chemical Methods SW-846, Method 9040C and 9045D.

21.4 ALS Environmental Quality Assurance Manual Revision (current version)

PERCENT MOISTURE

SW846 3550C

SOPID:	HN-WC-018	Rev. Number:	R06	Effective Date:	03/01/2018

Approved By: _____ Date: 2-20-18
Department Supervisor – Jen Jones ~~Grzan~~

Approved By: _____ Date: 2/19/18
Quality Assurance – Chad Stoike

Approved By: _____ Date: 2/20/18
Laboratory Director – Jeff Glaser

Archival Date: _____ Doc Control ID#: _____ Editor: _____

PROCEDURAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR 24 ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

Signature	Title	Date
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Signature	Title	Date

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PERCENT MOISTURE

1) Scope and Applicability

1.1 This SOP is used to determine percent moisture in solid and semi-solid samples such as soil, sediments, and sludge.

1.2 This SOP is a based upon and compliant with SW846 Method 3550C, Section 11.2.

2) Summary of Procedure

2.1 A well-mixed sample is evaporated in a weighed dish and dried to a constant weight in an oven at a temperature of 105°C. The increase in weight over that of the empty dish represents the total solids. Percent moisture is determined by calculation utilizing the total solids data.

3) Definitions

3.1 DI: Deionized reagent water, meeting purity characteristics of ASTM Type II or better.

3.2 Matrix: The component or substrate (e.g. surface water, groundwater, soil) containing the analyte of interest.

3.3 Method Blank: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing and carried through the complete sample preparation and analytical procedure.

3.4 Laboratory Control Sample (LCS): A clean, analyte-free matrix spiked with a known amount of target analyte(s) and carried through the complete preparation/analytical procedure.

3.5 Solids: The term "solids" refers to matter that is suspended or dissolved in water or wastewater.

3.6 Total Solids: Total solid is the term applied to the material residue left in the vessel after evaporation of a sample and its subsequent drying in an oven at a defined temperature.

3.7 Percent Moisture: Percent moisture is the term applied to the material evaporated from the sample during the drying process.

4) Health and Safety Warnings

4.1 Lab Safety

4.1.1 Due to various hazards in the laboratory, safety glasses, disposable gloves, and laboratory coats or aprons must be worn when working with unknown samples. In addition, heavy-duty gloves and a face shield are recommended when dealing with toxic, caustic, and/or flammable chemicals.

4.1.2 The toxicity or carcinogenicity of each reagent used has not been precisely defined. However, each chemical used must be treated as a potential health hazard and exposure reduced to the lowest possible level. The laboratory maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets (MSDS) is available to all personnel involved in these analyses.

4.2 Waste Disposal

4.2.1 Procedures for sample disposal are documented in SOP HN-SAF-001, *Waste Disposal Procedures.*

4.2.2 Samples must be disposed according to Federal, State, and local regulations.

4.3 Pollution Prevention

4.3.1 The quantities of chemicals purchased, when possible, must be based on the expected usage during its shelf life.

4.3.2 Standards and reagents must be prepared in volumes consistent with laboratory use to minimize the volume of expired standards or reagents to be disposed.

5) Cautions

5.1 Exercise caution when removing samples from the ovens, as they will be hot.

5.2 Tongs or heat resistant gloves must be used for the handling of hot containers.

6) Interferences

6.1 Weigh samples in an efficient and timely manner. Wet samples tend to lose weight by evaporation, and after drying or ignition, residues are often very hygroscopic.

7) Personnel Qualifications and Responsibilities

7.1 General Responsibilities – This method is restricted to use by or under the supervision of analysts experienced in the method.

7.2 Analyst – It is the responsibility of the analyst(s) to:

7.2.1 Read, understand, and follow this SOP as written.

7.2.2 Produce contractually compliant data that meets all quality requirements using this procedure and the Data Reduction, Review and Validation SOP (HN-QS-009).

7.2.3 Complete the required demonstration of proficiency before performing this procedure without supervision.

7.2.4 Create and populate a data entry batch in LIMS for review by the Supervisor.

7.3 Section Supervisor – It is the responsibility of the section supervisor to:

7.3.1 Ensure that all analysts have the technical ability and have received adequate training required to perform this procedure.

7.3.2 Ensure analysts have completed the required demonstration of proficiency before performing this procedure without supervision.

7.3.3 Produce contractually compliant data that meets all quality requirements using this procedure and the Data Reduction, Review and Validation SOP (HN-QS-009).

7.4 Project Manager – It is the responsibility of the Project Manager to:

7.4.1 Ensure that all contractual requirements for a client requiring this procedure are understood prior to initiating this procedure for a given set of samples.

8) Sample Collection, Handling, and Preservation

8.1 Sample collection bottles/jars – plastic or glass, approximately 1L or 4 oz. These are purchased by the laboratory and meet EPA specifications for sample containers.

8.2 Preserve the samples with refrigeration at 4±2°C from the time of collection until analysis is performed.

8.3 Samples should be placed in airtight containers immediately upon collection and maintained in the airtight container until analysis in order to minimize gains or losses in moisture from the atmosphere.

8.4 The holding time from sample collection to analysis shall not exceed 14 days.

9) Equipment and Supplies

9.1 Desiccators (with indicating desiccant)

9.2 Analytical balance – capable of weighing to the nearest 0.0001g

9.3 Drying oven at 104 ± 1°C

9.4 Aluminum weighing dishes with tabs

9.5 Crucibles and covers

9.6 Spatulas

9.7 Top-loading balance – capable of weighing to the nearest 0.01g

9.8 Tongs

9.9 Heat Resistant Gloves

10) Standards and Reagents

10.1 DI water – ASTM Type II or better

10.2 Sand – Ottawa Sand, purchased from an outside vendor and dried for at least 1 hour prior to use

11) Method Calibration

11.1 Balances must be checked daily with Class "S" weights appropriate for the range of use and recorded in the balance calibration logbook for each balance.

11.2 Daily oven temperatures must be recorded on the temperature monitoring logs (as read from the thermometer).

12) Sample Preparation/Analysis

12.1 Preparation of the evaporating dishes:

12.1.1 Dishes are dried at 104 ± 1°C for 1 hour and stored in a desiccator prior to use.

12.2 Weigh and record the weight of the aluminum weighing dish in the computer-generated worksheet. Tare the balance with the dish still on it after recording the initial weight.

12.3 Homogenize sample using formal subsampling techniques SOP (HN-QS-008), making sure to decant any standing water on top of the sample prior to processing.

12.4 Weigh out 5-10g of thoroughly homogenized sample into an aluminum weighing dish.

12.5 Record the wet weight of the sample on the computer-generated worksheet.

12.6 Dry the sample overnight at 104± 1°C.

12.6.1 Altered drying times can be used for quick turn analyses, however samples must be in the oven for a minimum of 1 hour and go through the re-dry process as described in section 12.9.

12.7 Remove the sample from the oven and cool in a desiccator for at least 1 hour.

12.8 Weigh and record the weight on the computer-generated worksheet.

12.9 If the samples were not dried in the oven overnight, repeat drying cycle until a constant weight is obtained, or until the weight change is < 4% or 0.5mg (whichever is smaller) of the previous reading.

12.10 Percent moisture values are determined by calculation using the data generated by performing the total solids test portion.

13) Troubleshooting

13.1 N/A

14) Data Acquisition

14.1 All data must be recorded on the computer generated Moisture Spreadsheet.

14.2 The computer-generated worksheets must be initialed, dated, and retained in a designated monthly folder.

15) Calculation, and Data Reduction Requirements

15.1 Calculations:

15.1.1 $$\% \text{ Total Solids} = \frac{\text{dry weight}}{\text{wet weight}} \times 100$$

15.1.2 % Moisture = 100 - % Total Solids

15.1.3 Relative Percent Deviation (RPD)

$$\% \text{ RPD} = \frac{[(SR_1 - SR_2)]}{½ (SR_1 + SR_2)} \times 100$$

Where:

SR_1 = sample result for replicate 1
SR_2 = sample result for replicate 2

16) Quality Control, Data Assessment and Corrective Action

16.1 Method Blank

16.1.1 Analyze a method blank with each analytical batch of 20 or fewer samples.

16.1.2 The analyzed concentration of analyte in the method blank must be < PQL.
16.1.3 The blank must be subject to the same procedural steps as a sample.
16.1.4 Preparation:

16.1.4.1 Carry an empty weighing dish through the entire drying cycle.

16.2 Duplicates

16.2.1 Sample duplicates must be processed at a 10% frequency
16.2.2 The RPD between duplicate analyses must meet precision performance criteria as outlined in the applicable LIMS test code.
16.2.3 Corrective Action:

16.2.3.1 If the RPD fails to meet the precision performance criteria as outlined in the LIMS test code, narrate that the RPD was not acceptable.
16.2.3.2 Turn off the parent sample and re-run for confirmation of moisture result.

16.3 Laboratory Control Sample (LCS)

16.3.1 Analyze a LCS with each analytical batch of 20 or fewer samples.
16.3.2 Recovery of the LCS must meet accuracy performance criteria as outlined in the applicable LIMS test code.
16.3.3 Preparation: Weigh out a portion of the pre-dried Ottawa Sand (Section 10.2) and carry the sample through the entire drying cycle.
16.3.4 Results for the LCS are reported in the terms of percent solids.

17) Data Records Management

17.1 All samples must be recorded in the applicable analytical logbook or electronic record.

17.2 All hardcopy and electronic records must be maintained for a period of no less than 10 years.

17.3 All final reports must be processed according to the guidelines documented in SOP HN-ADM-003, *Work Order Reporting*. Any subsequent revisions to previously finalized reports must be processed according the guidelines documented in SOP HN-QS-002, *Report Revisions*.

18) Quality Assurance and Quality Control

18.1 After analysis, the analyst must review raw data for accuracy, completeness, and notate any noted anomalies.

18.2 Each batch analytical batch must be initialed and dated by the primary analyst.

18.3 Each batch must be peer reviewed by the department supervisor (or designee) prior to finalization in LIMS (SOP HN-QS-009, *Data Reduction, Review, and Validation*).

19) Contingencies for Handling Out of Control Data

19.1 When method required QC failures occur, the source of the QC failure must be determined, corrected and sample re-analysis carried out when possible.

19.2 When sample analysis cannot be repeated due to limitations on sample availability, or if reanalysis can only be performed after expiration of a sample hold time, the

reporting of data associated with failed QC data must be appropriately flagged and narrated to define the error effect upon the data quality.

19.3 All analysts must report sufficient comments in LIMS so that project management can sufficiently narrate failed QC and ensure data qualifiers (flags) are properly assigned to the reported data.

19.4 Nonconformance reports must be documented on the associated analytical data checklist.

19.5 If the non-conformances are indicative of systemic or procedural errors, a corrective action, in conjunction with QA, must be documented and issued.

20) Method Performance

20.1 Each analyst must demonstrate initial proficiency with sample preparation and/or analytical determination by generating 4 sets of data of acceptable accuracy and precision for target analytes in a clean matrix.

20.2 Each analyst must demonstrate ongoing proficiency annually with each sample preparation and/or analytical determination method by generating 4 sets of data of acceptable accuracy and precision for target analytes in a clean matrix or by passing performance in approved PT evaluations.

20.3 An MDL is not applicable to this procedure.

21) Summary of Changes

Table 21.1 Summary of Changes

Revision Number	Effective Date	Document Editor	Description of Changes
R02	7/1/12	CES	Formatting
R03	12/15/14	CES	Updated method reference to 2540G
R04	02/15/15	CES	Updated method reference to 160.3M with a holding time of 14 days.
R05	02/02/16	CES	Updated reference method to SW846 3550C. Updated procedure for re-dry of samples only if not dried "overnight" per 3550C section 11.2.
R06	3/1/18	CES	Added section 16.2.3 - Corrective Action for failed RPD.

22) References and Related Documents

22.1 U.S. Environmental Protection Agency, "Method 3550C Ultrasonic Extraction", Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update IV, Revision 3, February 2007.

22.2 U.S. Environmental Protection Agency, Methods for Chemical Analysis of Waters and Wastes, EPA/600/4-79-020, Method 160.3.

22.3 ALS Environmental Quality Assurance Manual, Revision (most current).

ALS Standard Operating Procedure

DOCUMENT TITLE:	ULTRASONIC EXTRACTION
REFERENCED METHOD:	SW 846 3550C
SOP ID:	HN-EXT-013
REV. NUMBER:	R03
EFFECTIVE DATE:	08/31/2016

ULTRASONIC EXTRACTION

SW846 3550B

SOPID:	HN-EXT-013	Rev. Number:	R03	Effective Date:	08/31/2016

Approved By: _____ Date: 8/1/16
Department Supervisor

Approved By: _____ Date: 8/4/16
Operations Manager

Approved By: _____ Date: 7/29/16
QA Manager

Approved By: _____ Date: 8/1/16
Laboratory Director

Archival Date:	_____	Doc Control ID#:	_____	Editor:	_____

PROCEDURAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR 24 ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

Signature	Title	Date
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Signature	Title	Date

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ULTRASONIC EXTRACTION

1) Scope and Applicability

1.1 This method is a procedure for extracting nonvolatile and semi-volatile organic compounds from solids as soils, sludges, and wastes. The ultrasonic extraction process ensures intimate contact of the sample matrix with the extraction solvent. This SOP references SW-846 Method 3550C.

1.2 This method is applicable to the isolation and concentration of water-insoluble and slightly water soluble organics in preparation for a variety of chromatographic procedures.

1.3 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

1.4 A solvent dilution is used for oily samples prior to clean-up and/or analysis.

2) Summary of Procedure

2.1 The solid sample is mixed with baked HydroMatrix in a 250 ml beaker and extracted using an appropriate solvent (see Table 10.3) and ultrasonic extractor.

2.2 The extract is then dried, concentrated (if necessary), and, as necessary, exchanged into a solvent compatible with the cleanup or determinative step being employed (see Table 10.3).

2.3 If the sample is oil, then weigh one gram of sample into a 10 mL Class A volumetric and make to volume with the appropriate solvent.

3) Definitions

3.1 Laboratory Control Sample (LCS): An analyte-free matrix spiked with known concentrations of all target analytes. This is used to evaluate and document laboratory method performance.

3.2 Matrix: The component or substrate (e.g., surface water, groundwater, soil) which contains the analyte of interest.

3.3 Matrix Spike (MS): An aliquot of background sample spiked with a known concentrations of all target analytes. The spiking occurs prior to sample preparation and analysis. A matrix spike is used to assess the bias of a method in a given sample matrix.

3.4 Matrix Spike Duplicate (MSD): A duplicate aliquot of the background sample spiked with a known concentrations of all target analytes. Spiking occurs prior to sample preparation and analysis. The MS/MSD pair are used to assess precision and bias of a method in a given sample matrix.

3.5 Method Blank: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

3.6 Limit of Quantitation (LOQ): The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ is also referred to as the method quantitation limit (MQL) or the reporting limit (RL).

3.7 Limit of Detection (LOD): an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory-dependent.

3.8 Method Detection Limit (MDL) study: the procedure, as described in 40CFR part 136, for determining the LOD based on statistical analysis of 7 low-level replicate spikes.

4) Health and Safety Warnings

4.1 Lab Safety: Due to various hazards in the laboratory, safety glasses and laboratory coats or aprons must be worn at all times while in the laboratory. In addition, gloves and a face shield should be worn when dealing with toxic, caustic, and/or flammable chemicals.

4.2 Chemical Hygiene: The toxicity or carcinogenicity of each reagent used has not been precisely defined; however, each chemical used should be treated as a potential health hazard. Exposure to laboratory reagents should be reduced to the lowest possible level. The laboratory maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets (MSDS) is available to all personnel involved in these analyses.

4.3 Waste Management: The principal wastes generated by this procedure are the method-required chemicals and standards. It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is required. Laboratory procedures in SOP HN-SAF-001, Waste Disposal Procedures, must be followed.

4.4 Pollution Prevention: The materials used in this method pose little threat to the environment when recycled and managed properly. The quantities of chemicals purchased should be based on the expected usage during its shelf life. Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards or reagents to be disposed.

5) Cautions

5.1 All glassware must be clean and rinsed thoroughly with solvent prior to transferring from one container to the next. For glassware cleaning procedures, refer to SOP HN-GEN 003.

5.2 Exposure to plastic materials must be minimized the greatest extent possible, as they are a source of phthalate (an analyte of concern) contamination. Gloves, tubing, and other laboratory materials must be free of these materials.

5.3 When the volume of solvent is reduced below 1ml, semivolatile analytes may be lost. Care must be taken when reducing volume to 1 ml.

5.4 Never allow the solvent level of any samples to go dry. When the solvent goes dry, the extraction process must be repeated.

6) Interferences

6.1 The decomposition of some analytes has been demonstrated under basic extraction conditions. Organo-chlorine pesticides may de-chlorinate, phthalate esters may exchange, and phenols may react to form tannates.

6.2 Method interferences may be caused by contaminants in solvents, reagents, glassware, and sample processing hardware. These contaminants lead to discrete artifacts or to

elevated baselines in gas chromatograms. These materials must be routinely demonstrated to be free from interferences under the sample preparation and analysis conditions by analyzing instrument blanks and method blanks.

6.3 Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Because common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates are minimized when the use of plastic materials is avoided.

6.4 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the site being sampled. The cleanup procedures in this method must be used to remove such interferences in order to achieve the Method Quantitation Limits (MQL).

7) Personnel Qualifications and Responsibilities

7.1 General Responsibilities - This method is restricted to use by or under the supervision of analysts experienced in the method.

7.2 Analyst - It is the responsibility of the analyst(s) to:

- 7.2.1 Each must read and understand this SOP and follow it as written. Any deviations or non-conformances must be documented and submitted to the QA Manager for approval.
- 7.2.2 Produce method compliant data that meets all quality requirements using this procedure and the Data Reduction, Review and Validation SOP (HN-QS-009).
- 7.2.3 Complete the required initial demonstration of proficiency before performing this procedure without supervision.
- 7.2.4 Complete an ongoing demonstration of proficiency annually when continuing to perform the procedure.
- 7.2.5 The analysts must submit data for peer or supervisor review.

7.3 Section Supervisor - It is the responsibility of the section supervisor to:

- 7.3.1 Ensure that all analysts have the technical ability and have received adequate training required to perform this procedure.
- 7.3.2 Ensure analysts have completed the required initial demonstration of proficiency before performing this procedure without supervision.
- 7.3.3 Ensure analysts complete an ongoing demonstration of proficiency annually when continuing to perform the procedure.
- 7.3.4 Ensure analysts produce method compliant data that meet all quality requirements using this procedure and the Data Reduction, Review and Validation SOP.

7.4 Project Manager - It is the responsibility of the Project Manager to ensure that all method requirements for a client requesting this procedure are understood by the laboratory prior to initiating this procedure for a given set of samples.

7.5 QA Manager: The QA Manager is responsible for

- 7.5.1 Approving deviations and non-conformances
- 7.5.2 Ensuring that this procedure is compliant with method and regulatory requirements,
- 7.5.3 Ensuring that the analytical method and SOP are followed as written through internal method and system audits.

8) Sample Collection, Handling, and Preservation

8.1

SEMIVOLATILE ORGANICS AND ORGANOCHLORINE PESTICIDES/PCBS			
Sample Matrix	Container	Preservative	Holding Time
Concentrated Waste Samples	125- mL widemouth glass with Teflon-lined lid	None	Extraction 14 days/Analysis 40 days
Solid Samples	250-mL widemouth glass container with Teflon-lined lid	Cool to 4°C	Extraction 14 days/Analysis 40 days

9) Equipment and Supplies

9.1 Ultrasonic extraction devise w/appropriately sized horn (3/4")

9.2 250 ml beakers

9.3 500 ml Kuderna Danish concentration apparatus

9.4 Boiling chips - Solvent-extracted, approximately 10/40 mesh (silicon carbide or equivalent).

9.5 Vials - Glass, 2ml and 12ml capacity, with Teflon™ lined screw or crimp top.

9.6 Disposable glass Pasteur pipet & bulb.

9.7 Drying oven- capable of maintaining 105 degrees Celsius.

9.8 Top Loading balance- capable of weighing to 0.01 g.

9.9 Spatula

9.10 Syringes, various sizes - 500ul, 1ml, etc. (perform annual calibration checks on syringes)

9.11 N-Vap concentration unit

9.12 Centrifuge (~3000 rpm max) or equivalent

9.13 Turbo-Vap concentration unit

9.14 200mL Turbo-Vap concentration tubes

10) Standards and Reagents

10.1 HPLC, Pesticide, and other such high purity solvents shall be used for all tests. Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall confirm to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Reagents should be stored in glass to prevent the leaching of contaminants from plastic containers.

10.2 HydroMatrix. Purify by heating at 400 degrees Celsius for four hours or by pre-cleaning with methylene chloride.

10.3 Soil/sediment and aqueous sludge samples shall be extracted using either of the following solvent systems:

Table 10.3 – Method Extraction Solvent Systems		
Determinative Method	Initial Extraction Solvent	Exchange Solvent
Organochlorine Pesticides and PCBs (8081A and 8082)	80% Hexane/20% Acetone	Hexane
Semi-volatile Organics (8015D, 8270D & 8310),	1:1 Methylene chloride/Acetone	None

10.4 Spiking Solutions:

10.4.1 PCB Surrogate Spike @ 1 ug/ml

10.4.2 PCB Matrix Spike @ 5 ug/ml

10.4.3 Pesticide Surrogate Spike @ 1 ug/ml

10.4.4 Pesticide Matrix Spike @ 1 ug/ml

10.4.5 DRO/ORO Matrix Spike @ 50,000ug/ml

10.4.6 Semi-Volatile and DRO/ORO Surrogate Spike @ 100 ug/ml

10.4.7 Semi-Volatile Matrix Spike @ 40 ug/ml

10.5 Spiking solutions should be prepared as needed from stock standards according to the applicable SOP. Solutions shall be stored refrigerated and protected from light. Stock standards should be replaced after 1 year or sooner if a manufacturer expiration date has been reached. Prepared intermediate standards should be replaced every 6 months or sooner if a component expires sooner.

10.6 Reagent Preparation Records: Record Stock Standards in the Chemical Inventory Log-book and record all standards prepared in the Standard Preparation Logbook according to SOP HN-QS-001. Label standards appropriately so that they can be easily cross-referenced to other records.

11) Method Calibration

11.1 Calibrate support equipment according to appropriate calibration schedules.

11.2 The sonicator horn must be tuned prior to use, according to manufacturer instructions.

12) Sample Preparation/Analysis

12.1 Decant and discard any water layer on a sediment sample. Discard any foreign objects such as sticks, leaves, and rocks.

12.2 Waste samples: Samples consisting of multiple phases must be prepared by the phase separation method. This extraction procedure is for solids only.

12.3 Gummy, fibrous or oily materials not amenable to grinding should be cut, shredded, or otherwise reduced in size to allow mixing and maximum exposure of the sample surfaces for the extraction.

12.4 Ultrasonic Extraction:

12.4.1 Homogenize the sample according to the procedure outlined in HN-QS-008, Sample Homogenization.

12.4.2 For semi-volatile analyses, use 30 g (weighed to the nearest 0.1 g) of the solid sample. For DRO/ORO analyses, use 25g. For PCB and Pesticide analyses, use 15 g.

12.4.3 Spike all samples with appropriate surrogates. Spike all LCS and MS/MSD samples with the appropriate spike mix prior to adding drying agent. See Table 21.7 for spike volumes.

12.4.4 In a 250 ml beaker, blend the sample with sufficient Hydromatrix to create a dry, free-flowing mixture.

12.4.5 Add approximately 60 ml of the extraction solvent.

12.4.6 Place in the ultrasonic apparatus and position such that the horn is below the solvent but above the sample.

12.4.7 Sonicate for 3 minutes at an amplitude of 100. Visually inspect during the extraction procedure to ensure sample/solvent agitation.

12.4.8 Remove from the ultrasonic apparatus and filter the solvent through filter paper into a 500 ml K-D equipped with a 10 ml concentrator tube. A 200ml Turbo-vap concentration tube may be substituted for the K-D if that concentration technique will be utilized.

12.4.9 Repeat Sections 12.4.5 through 12.4.8 two additional times.

12.4.10 After three iterations, rinse the filter with approximately 10 ml of the extraction solvent.

12.4.11 Add 1-2 boiling chips to the concentration apparatus and install a 3-ball Snyder column if using the K-D.

12.4.12 Proceed to Section 12.5 or 12.7, dependent on which concentration technique is used.

12.5 K-D Concentration Technique:

12.5.1 Place the K-D setup in the hot water bath. Water bath must be in a hood or have a solvent recovery system attachment.

12.5.2 Concentrate the sample to approximately 5 ml. Ensure that the water bath is maintained at a temperature of 90° C when concentrating Methylene chloride/Acetone.

12.5.3 If solvent exchange is required, add approximately 10 ml of hexane (or appropriate solvent) and re-concentrate to approximately c ml.

12.5.4 Remove K-D assembly from water bath and allow to cool.

12.5.5 Prior to removing concentration tube, carefully wipe the connecting joint with a Chem-Wipe and visually inspect to ensure the absence of any water.

12.5.6 Carefully remove the concentrator tube and transfer to the N-Vap Blow-Down Unit.

12.6 N-Vap Blow Down

12.6.1 Blow down sample to approximately 0.5-1.0 ml with a gentle stream of Nitrogen.

12.6.2 If Pest/PCB analysis, quantitatively transfer the extract to an appropriately labeled vial and bring to a final volume of 5 ml with hexane.

12.6.3 If extract is for Semi-Volatile analysis, quantitatively transfer to an appropriately labeled vial and bring to a final volume of 1.0 ml with methylene chloride.

12.6.4 Transfer the finalized extracts to the appropriate analytical section for storage and/or analysis.

12.7 Turbo Vap™ Concentration Technique:

12.7.1 If PCB/Pest analysis:

12.7.1.1 Blow down to approximately 3.0 mL and quantitatively transfer to an appropriately labeled vial.
12.7.1.2 Bring to a final volume of 5.0 mL with Hexane.

12.7.2 If extract is TPH sample:

12.7.2.1 Blow down sample to approximately 0.75 mL.
12.7.2.2 Quantitatively transfer to an appropriately labeled vial.
12.7.2.3 Bring to a final volume of 1.0 mL with 1:1 Methylene Chloride:Acetone

12.7.3 Transfer the finalized extracts to the appropriate analytical section for storage and/or analysis.

12.8 Sulfuric Acid Clean up Procedure for PCBs (SW 3665A): For field samples exhibiting high levels of hydrocarbon contamination, a sulfuric acid cleanup may be used. If so, all batch extracts (blanks, QC samples and client samples) must be processed with the cleanup.

12.8.1 Using a disposable glass dispensing pipette and transfer about 3.0-mLs of the hexane extract to a 10-mL vial. Carefully add 3-mLs of concentrated sulfuric acid. CAUTION: Make sure that there is no exothermic reaction or evolution of gas prior to proceeding.
12.8.2 Cap the vial tightly and vortex for one minute. A vortex must be visible in the vial. CAUTION: Stop the vortexing immediately if the vial leaks. AVOID SKIN CONTACT, SULFURIC ACID BURNS.
12.8.3 Allow the phases to separate for at least 1 minute. Examine the top (hexane) layer; it should not be highly colored nor should it have a visible emulsion or cloudiness. Separation may also be performed using a table-top clinical centrifuge, operating for one minute on Speed #3.
12.8.4 If a clean phase separation is achieved, proceed to Sec. 12.8.6.
12.8.5 If the hexane layer is colored or an emulsion persists for several minutes, remove the hexane layer from the top of the sulfuric acid layer and transfer it to another clean vial containing a 3-mL portion of the clean concentrated sulfuric acid solution. Repeat the acid cleanup step, beginning at Sec. 12.8.1. This step may be repeated twice as needed. Any remaining acid layers from the bottom of the cleanup vials must be disposed of properly.
12.8.6 Transfer the hexane layer to a clean 2-mL autosampler vial. Care must be taken not to include any of the acid when transferring the hexane layer to the clean vial, as it can cause damage to the analytical instrumentation.

13) Troubleshooting

13.1 Good technique is important to obtain consistently acceptable LCS, MS, and MSD recoveries. Good housekeeping and thorough equipment and glassware cleaning are a must to prevent sample contamination. Refer to Glassware Cleaning SOP.

14) Data Acquisition

14.1 Sample preparation information is logged into LIMS by starting a prep batch and entering the pertinent information. This information is used in calculating the analytical results.

15) Calculation, and Data Reduction Requirements

15.1 QC Calculations: LIMS calculates the percent recovery for various QC samples (MS, MSD, LCS) according to the following equations:

15.1.1 % Recovery, %R (for MS and MSD Samples)

$$\%R = \frac{(SSR - SR)}{SA} \times 100$$

Where:
SSR = Spiked Sample Result (mg/L or mg/kg).
SR = Sample Result (unspiked).
SA = Spike Amount Added (mg/L or mg/kg).

15.1.2 % Recovery, %R (for standards and LCS)

$$\%R = \frac{(SSR)}{SA} \times 100$$

Where:
SSR = Spiked Sample Result (mg/L or mg/kg).
SA = Spike Amount Added (mg/L or mg/kg).

15.1.3 RPD (for precision or duplicate evaluation)

$$RPD = \frac{|SR_1 - SR_2|}{½(SR_1 + SR_2)} \times 100$$

Where:
SR_1 = Sample result for duplicate 1.
SR_2 = Sample result for duplicate 2.

16) Quality Control, Acceptance Criteria and Corrective Action

16.1 Holding Time: Extract solids/soils within 14 days of collection.

16.2 Method Blank

16.2.1 Frequency: One per batch of sample digestions (a batch = 20 or less commercial samples)
16.2.2 Criteria: All analytes of interest should be less than ½ the MRL and must be less than reporting limit. When analytes are detected above the reporting limit, a non-conformance report must be issued.
16.2.3 Corrective action: Find source of contamination and correct the problem. Re-extract all QC samples and batch samples that have detection above the reporting limit.

16.3 Laboratory Control Samples (LCS)

16.3.1 Frequency: One per batch of 20 or less sample extractions.
16.3.2 Criteria: See applicable analytical SOP.

16.4 Matrix Spike / Matrix Spike Duplicate (MS/MSD)

16.4.1 Frequency: Matrix spikes will be analyzed on a frequency of one spike for each 20 samples analyzed. If fewer than 20 samples are in a batch, at least one spike will be included.

16.4.2 Criteria: See applicable analytical SOP.

16.5 Surrogate Standards must be added to all samples and QC samples.

16.6 For more complete information on data assessment and corrective action, refer to each determinative method SOP. The analyst will make the primary decisions concerning assessment and correction action.

16.7 Deviations and non-conforming events must be documented using a Nonconformance Corrective Action Report (NCAR) or as an Exception Report item on the laboratory review checklist. For mandatory QC failures (e.g. LCS), the NCAR must be submitted to the QA Manager via the NCAR database.

17) Data Records Management

17.1 All data is stored both electronically and hard copy for 10 years.

17.2 All analytical sequence IDs and standard preparation information must be recorded in the Run logbook. Hardcopy computer printouts of analytical sequences and raw data must be retained and initialed by the analyst (electronic initials are acceptable). To simplify standard tracking, analyst must attempt to use one lot of reagents and standards with each batch.

17.3 Complete all pertinent sections in the respective logbooks. If not-applicable then line out the section. "Z" out or "X" out all large sections of the worksheet that are not used. Make all corrections with single line through, date and initial. Make NO obliterations when manually recording data.

17.4 Logbooks are controlled. Never remove a page from a logbook. Completed logbooks are returned to the QA department when filled and no longer needed in the work area.

17.5 The effective date this SOP is the date in the header or last signature date, whichever is most recent

18) Contingencies for Handling Out of Control Data

18.1 When method required QC exceedances occur, in every case where sample data quality are affected, the source of the QC exceedance must be determined, corrected and sample reanalysis carried out when possible.

18.2 When affected sample analysis cannot be repeated due to limitations (i.e. sample availability, or if reanalysis can only be performed after expiration of a sample hold time), the reporting of data associated with exceeded QC data must be appropriately flagged and narrated. This documentation is necessary to define for the data user the effect of the error has upon the data quality of the results reported (e.g. E flag data indicate the result to be only an estimate).

18.3 All analysts must report sufficient comments in laboratory data review checklist for exceeded QC associated with sample results so that project management can further narrate and ensure data qualifiers (flags) are properly assigned to the reported data.

18.4 NCARs must be issued for QC system exceedances. Matrix interferences are reported using the analyte reporting comment section in LIMS or using the Laboratory Data review checklist.

19) Method Performance

19.1 Initial Demonstration of Proficiency- Each analyst must perform an initial demonstration of proficiency on a method and matrix basis with a successful analysis of four LCS where acceptable precision and accuracy are generated. The accuracy component must fall within LCS criteria. The precision component must be less than 20% for duplicate RPD data.

19.2 Method Detection Limits (MDLs) must be determined on an annual basis (at minimum) or whenever major modifications are performed.

20) Summary of Changes

Table 20.1 Summary of Changes

Revision Number	Effective Date	Document Editor	Description of Changes
R01	9/1/13	CES	New SOP
R02	1/15/16	CES	Addition of DRO/ORO and Turbo-Vap concentration technique.
R03	8/31/16	CES	Section 12.4.1 – 12.4.3 updated for sequence of events.

21) References and Related Documents

21.1 U.S. Environmental Protection Agency, "Method 3500C, Organic Extraction and Sample Preparation", Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update IV, February, 2007.

21.2 U.S. Environmental Protection Agency, "Method 3550C Ultrasonic Extraction", Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update IV, February, 2007.

21.3 U.S. Environmental Protection Agency, "Method 8000B Determinative Chromatographic Separations", Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update III, June 13, 1997.

21.4 U.S. Environmental Protection Agency, "Method 3665A, Sulfuric Acid Cleanup", Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Revision 1, December 1996.

21.5 US Army Corp of Engineers Shell Document, EM 200-1-3, Feb '01.

21.6 ALS Environmental Quality Assurance Manual, Revision (most current)

21.7 Table 21.7 – Surrogate and Analyte Spike Additions

Table 21.7 - Surrogate & Analyte Spike Additions

Analysis	*Final Volume*	*Surrogate Addition*	*Spike Addition*
PCBs	5 ml	0.5 ml @ 1 ug/ml	2.5 ml @ 5 ug/ml
Pesticides	5 ml	0.5 ml @ 1 ug/ml	0.5 ml @ 1 ug/ml
SVOA	1 ml	0.5 ml @ 100 ug/ml	0.5 ml @ 40 ug/ml
DRO/ORO	1 ml	0.5 ml @ 100 ug/ml	0.1 ml @ 50,000 ug/ml

ALS Standard Operating Procedure

DOCUMENT TITLE:	MICROWAVE EXTRACTION OF ORGANIC CONSTITUENTS IN SOIL
REFERENCED METHOD:	SW846 3546
SOP ID:	HN-EXT-016
REV. NUMBER:	R03
EFFECTIVE DATE:	06/06/2017

ALS Environmental

MICROWAVE EXTRACTION OF ORGANIC CONSTITUENTS IN SOIL

SW846 3546

SOPID:	HN-EXT-016	Rev. Number:	R03	Effective Date:	06/06/2017

Approved By: _____ Date: 6/6/17
Department Supervisor

Approved By: _____ Date: 6/6/17
Operations Manager

Approved By: _____ Date: 6/6/17
Quality Assurance

Approved By: _____ Date: 6/6/17
Laboratory Director

Archival Date: _____ Doc Control ID#: _____ Editor: _____

PROCEDURAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR 24 ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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MICROWAVE EXTRACTION

1) Scope and Applicability

1.1 This method is a procedure for extracting nonvolatile and semivolatile organic compounds from solids as soils, sediments, sludge, and wastes.

1.2 This method is applicable to the isolation and concentration of water-insoluble and slightly water-soluble organics in preparation for a variety of chromatographic procedures.

1.3 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

1.4 This SOP is based upon and references SW-846 Method 3546.

2) Summary of Procedure

2.1 The solid sample is mixed with drying agent (hydro-matrix, sodium sulfate, etc.), placed in a PFA vessel, and extracted using an appropriate solvent (see Table 10.4) in a laboratory microwave oven. Elevated pressure and temperature create the appropriate extraction conditions.

2.2 The extract is then concentrated, and, as necessary, exchanged into a solvent compatible with the cleanup or determinative step being employed. (see Table 10.4).

3) Definitions

3.1 Laboratory Control Samples (LCS): A known matrix spiked with compound(s) representative of the target analytes. This is used to evaluate and document laboratory method performance.

3.2 Matrix: The component or substrate (e.g., surface water, groundwater, and soil) that contains the analyte of interest.

3.3 Matrix Spike Samples (MS/MSD): An aliquot of sample spike with a known concentration of target analytes. The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

3.4 Method Bank: An analyte free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

3.5 Stock Standard Solution: A concentrated solution containing certified standards that are the target or method analytes. Stock standard solutions are used to prepare the calibration and other QC standards (LCS, MS, and surrogate spiking standards, etc.).

3.6 Surrogate: An organic compound which is similar to the target analytes in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.

3.7 Preparation Batch: A defined set of one to 20 client samples of the same matrix, meeting the batch definition criteria, and prepared for analysis within an 8 hour

working shift. The preparation batch must also contain the required determinative method defined QC samples.

4) Health and Safety Warnings

4.1 Lab Safety

4.1.1 Due to various hazards in the laboratory, safety glasses, disposable gloves, and laboratory coats or aprons must be worn when working with unknown samples. In addition, heavy-duty gloves and a face shield are recommended when dealing with toxic, caustic, and/or flammable chemicals.

4.1.2 The toxicity or carcinogenicity of each reagent used has not been precisely defined. However, each chemical used must be treated as a potential health hazard and exposure reduced to the lowest possible level. The laboratory maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets (MSDS) is available to all personnel involved in these analyses.

4.2 Waste Disposal

4.2.1 Procedures for sample disposal are documented in SOP HN-SAF-001, *Waste Disposal Procedures*.

4.2.2 Samples must be disposed according to Federal, State, and local regulations.

4.3 Pollution Prevention

4.3.1 The quantities of chemicals purchased, when possible, must be based on the expected usage during its shelf life.

4.3.2 Standards and reagents must be prepared in volumes consistent with laboratory use to minimize the volume of expired standards or reagents to be disposed.

4.4 All solvents and reagents used in this procedure shall be handled in a fume hood using chemical resistant gloves. Lab coat and safety glasses shall also be worn.

4.5 This procedure employs the use of methylene chloride, a suspected human carcinogen.

4.6 The microwave exhaust line must be routed into a fume hood.

4.7 The extraction vessels will be under a pressurized state until cooled back down to room temperature. Vessels shall be allowed to sufficiently cool prior to opening.

5) Cautions

5.1 All labware must be clean and rinsed thoroughly with solvent prior to transferring from one container to the next. For glassware cleaning procedures, refer to SOP HN- GEN-003.

5.2 Exposure to plastic materials should be minimized to the greatest extent possible, as they are a source of phthalate (an analyte of concern) contamination. Gloves, tubing and other laboratory materials should be free of these materials.